



MPI
RESEARCH

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State College, Pennsylvania 16801 USA
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Analytical Report

**PFOA and PFOS Analysis of Wild Turkey Muscle and Liver Samples by
LC/MS/MS**

MPI Report No. L0019897

Testing Laboratory

MPI Research, Inc.
3058 Research Drive
State College, PA 16801

Requester/Project Manager

Dena Haverland
Dalton Utilities
PO BOX 869
Dalton, GA 30722
Phone: 706-529-1010

1 Introduction

Results are reported for the analysis of turkey muscle and liver samples received at MPI Research from Dalton Utilities. The MPI Research study number assigned to the project is L0019897. Table I lists the target analytes quantitated for the samples.

Table I. Target Analytes for Quantitation

Compound Name	Acronym
Perfluorooctanoic Acid	C8 Acid or PFOA
Perfluorooctanesulfonate	C8 Sulfonate or PFOS

2 Sample Receipt

Four samples were received from Dena Haverland at Dalton Utilities for this study. The samples were collected on December 16, 2009. The samples arrived on December 22, 2009 via Fedex and were logged in under MPI Research login number L0019897. The shipment was received frozen on dry ice. The samples were stored frozen at approximately -20°C from receipt until analysis. Chain-of-custody information is presented in Attachment A.

3 Methods - Analytical and Preparatory

3.1 Muscle and Liver Sample Preparation

- 3.1.1. Weigh 1 g of muscle or liver sample into a 50 mL disposable centrifuge tube and fortify, if appropriate. Add appropriate amount of internal standard solution.
- 3.1.2. Add water to the sample for a final volume of 10 mL. Cap tightly.
- 3.1.3. Homogenize sample using a tissuemizer for ~1 minute.
- 3.1.4. Transfer 1 mL of the sample using a disposable pipette into 15 mL disposable centrifuge tubes. Add 5 mL of ACN and shake for ~20 minutes on a wrist action shaker.
- 3.1.5. Centrifuge tubes at ~3000 rpm for ~5 minutes. Carefully decant supernatant into a 50 mL disposable centrifuge tube and add 35 mL of water.
- 3.1.6. Place the unconditioned SPE columns on the vacuum manifold. Condition the SPE columns by passing ~10 mL of methanol through the column followed by ~5 mL of water. The washes may be pulled through the SPE column using vacuum at a flow rate of ~1 drop/sec or may be allowed to pass through the column unaided. Discard all washes. Do not allow the column to dry.
- 3.1.7. Load the sample onto a conditioned SPE column. Discard the eluate. Any analyte residues will be trapped on the SPE column at this point.
- 3.1.8. Elute with 2 mL of methanol. Collect 2 mL of elute into a graduated 15 mL centrifuge tube.

Note: Post extraction dilutions were prepared in methanol.

3.2 Sample Analysis by LC/MS/MS

In High Pressure Liquid Chromatography (HPLC), an aliquot of extract is injected and passed through a liquid-phase chromatographic column. Based on the affinity of the analyte for the stationary phase in the column relative to the liquid mobile phase, the analyte is retained for a characteristic amount of time. Following HPLC separation, mass spectrometry provides a rapid and accurate means for analyzing a wide range of organic compounds. Molecules are ionized, fragmented, and detected. The ions characteristic of the compounds are observed and quantitated against external calibration standards.

An HP1100 system interfaced to an Applied Biosystems API 4000 LC/MS/MS was used to analyze the sample extracts for quantitation. A gradient elution through a Phenomenex Luna 3 μ C8(2) Mercury, 20 x 4.0 mm column was used for separation.

The following gradient was performed:

Mobile Phase (A): 2mM Ammonium Acetate in Water
Mobile Phase (B): Methanol

<u>Time</u>	<u>%A</u>	<u>%B</u>
0.0	90	10
0.5	90	10
2.0	10	90
5.0	10	90
5.1	0	100
6.0	0	100
6.1	90	10
10.0	90	10

The following parameters were used for operation of the mass spectrometer:

Parameter	Setting
Ionization Mode	Electrospray
Polarity	Negative
Transitions Monitored	413→369 (PFOA)
	413→219 (PFOA Confirmation)
	499→80 (PFOS)
	499→99 (PFOS Confirmation)
	415→370 (Internal Std. ¹³ C PFOA (m+2))
Gas Temperature	503→80 (Internal Std. ¹³ C PFOS (m+4))
	450°C

4 Analysis by LCMSMS

4.1 Calibration

For the muscle and liver sample analysis, a 6-point calibration curve was analyzed throughout the analytical sequence for PFOS. The calibration points were prepared at 0.1, 0.2, 0.5, 1.0, 2.0, 5.0 ng/mL (ppb) containing 1.0 ng/mL ^{13}C -PFOS (m+4).

The ratio of the analyte concentration to the IS concentration versus the ratio of the analyte instrument response (area) to the IS response (area) was plotted for each point. Using linear regression with 1/x weighting, the slope, y-intercept and coefficient of determination (r^2) were determined. A calibration curve is acceptable if $r^2 \geq 0.985$.

For the results reported here, calibration criteria were met. The calibration curves are included in the raw data in Attachment C.

4.2 Laboratory Control Spikes

Laboratory control spikes in the analytical set were prepared during each extraction set by adding a known concentration of the analyte to turkey muscle and liver controls. Laboratory control spikes are used to assess method accuracy. The laboratory control spikes must show recoveries between 70-130% or the data is rejected. For the results reported here, the laboratory control spikes were within the acceptable range. Laboratory control spike recoveries are given in Attachment B.

4.3 Matrix Spikes

A matrix spike was prepared for each sample by adding a known concentration of the target analyte to a sample. Matrix spikes are used to assess method accuracy in the matrix. The matrix spikes should show recoveries between 70-130%. For the results reported here, the matrix spike was within the acceptable range with the exceptions of:

4.4 Laboratory Duplicates

Each sample was prepared in duplicate and analyzed. Duplicate results are given along with the sample results in Attachment B.

5 Data Summary

Due to an interfering matrix peak at the 499→80 m/z transition, the 499→99 m/z confirmation transition was used for quantitation. The interfering matrix peak was not present at the 499→99 m/z confirmation transition.

Please see Attachment B for a detailed listing of the analytical results. For the muscle and liver samples the results are reported in parts per billion (ng/g) on an as-received basis.


6 Data/Sample Retention

Samples are disposed of 60 days after the report is issued unless otherwise specified by the project manager. All electronic data is archived on retrievable media and hard copy reports are stored in data folders maintained by MPI Research. Hardcopy data is stored for a minimum of five years. The client will be notified 30 days prior to the disposal of hardcopy data.

7 Attachments

- 7.1 Attachment A: Chain of Custody
- 7.2 Attachment B: Analytical Results
- 7.3 Attachment C: Raw Analytical Data

8 Signatures


Mark Neeley, Research Chemist Associate II

2-16-10
Date

Robert Zhu, Manager, Analytical Date

A



Mattawan (Corporate Headquarters)
54943 North Main Street
Mattawan, MI 49071-9399
(269) 668-3336 Phone
(269) 668-4151 Fax

State College
3058 Research Drive
State College, PA 16801
(814) 272-1039 Phone
(814) 231-1580 Fax

Login

Login Group: L0019897

Login #: 20011
Project: P0005380
Company Name: Dalton Utilities
Submitted By: Dena Haverland
Login Type: Immediate Receipt of Samples
Started: True
Date Start: 12/31/2009
Due Date: 01/10/2010
Login Initiated: 12/31/2009
Received By: Kyle, Matt
Spread Sample:
Label:
MPI SD/PI: Zhu, Xiang
Project Title/Type: PFOA AND PFOS ANALYSIS OF TURKEY BLOOD, MUSCLE AND LIVER / ROUTINE
Login Notes:

Conform COC Sample: True
Conform COC: True
Conform Sample: True
Conform Request: True

Packages / Containers

<u>Package</u>	<u>Carton</u>	<u>Date / Condition</u>	<u>Shipper / ID</u>	<u>Temp. Control/Temp.</u>	<u>Direction / Handled By</u>	
PK0022626		Received Date: 12/22/09 11:33 Package & Contents Uncompromised	FEDEX 8694 2057 8384	Dry Ice -79.0	RECEIVED Kyle, Matt	
<u>Container #</u>	<u>Gross Weight</u>	<u>pH</u>	<u>Container Type</u>	<u>Preservative</u>	<u>Mfg. Lot</u>	<u>Mfg. ID</u>
0473182	417.70 g		1 gallon ziploc bag	NONE		
C0473183	143.40 g		1/2 gallon ziplock bag	NONE		
C0473184	458.00 g		1 gallon ziploc bag	NONE		
C0473185	89.90 g		1/2 gallon ziplock bag	NONE		

Samples

Sample ID	Container	Matrix	System	System Matrix	Sample	Date Sampled	Date Due
L0019897-0001	C0473182	SOLID	Deer	Tissue	Wild Turkey #2 4yr Male - Muscle	12/16/2009	01/10/2010
L0019897-0002	C0473183	SOLID	Deer	Liver	Wild Turkey #2 4yr Male - Liver	12/16/2009	01/10/2010
L0019897-0003	C0473184	SOLID	Deer	Tissue	Wild Turkey #5 1yr Male - Muscle	12/16/2009	01/10/2010
L0019897-0004	C0473185	SOLID	Deer	Liver	Wild Turkey #5 1yr Male - Liver	12/16/2009	01/10/2010



[Login](#)

_____ M/2

Date/Time: 02/07/10 1356

MPI RESEARCH

MPI Research Contact: Daniel Wright

Send Report To:

Company: Dalton Utilities
 Address: 1200 VD Parrott JR Parkway, PO Box 869
 City, State, ZIP: Dalton, GA 30722-0869
 Attention: Dena Haverland
 Phone #: 706-529-1010
 Fax #: 706-529-1271
 Email: dhaverland@dutil.com
 Study/Job #: _____
 Signature/Date: _____
 Printed Name: _____

Sample Submittal

Please fax this form before sending samples.

Please send samples to shipping and receiving:
 3048 Research Drive, State College, PA 16801
 T: (814) 272-1039 • F: (814) 272-1019

Turnaround time (TAT) requirements:

Results Due Date: 30 days
 Preliminary Results Format: Verbal ☒ Email ☐ Fax
 Report Due Date: 30 days

Storage Conditions

Room temperature
 Refrigerator
☒ Freezer
 Ultra Low freezer
 Desiccated
 Lighting required

Stability (°C/%RH): _____

Stability time period: _____

Safety Information

Special handling: _____
 MSDS attached
 Controlled substance: _____
 HAZARDS: _____
 Please fill in the diamond HMIS/NFPA
 (0 4) if appropriate

Client ID#	Description	Lot/ Control #	Amt. Sent/ Weight	# of Bottles	Matrix	Date & Time	Tests Requested
1	Wild Turkey #2 4yr Male - Serum		3ml	2	Turkey	12/16/09 11:15am	PFOA/PFOS
2	Wild Turkey #2 4yr Male - Muscle		408gm	1 bag	Turkey	12/16/09 11:15am	PFOA/PFOS
3	Wild Turkey #2 4yr Male - Liver		131gm	1 bag	Turkey	12/16/09 11:15am	PFOA/PFOS
4	Wild Turkey #5 1yr Male - Serum		4ml	2	Turkey	12/16/09 12:00pm	PFOA/PFOS
5	Wild Turkey #5 1yr Male - Muscle		442gm	1 bag	Turkey	12/16/09 12:00pm	PFOA/PFOS
6	Wild Turkey #5 1yr Male - Liver		82gm	1 bag	Turkey	12/16/09 12:00pm	PFOA/PFOS
7							
8							
9							
10							

PO #: _____

Notes:

Relinquished by	Date	Time	Received by	Date	Time
<u>Daniel Wright</u>	<u>12/21/09</u>	<u>3:00pm</u>	<u>[Signature]</u>	<u>12/22/09</u>	<u>11:37</u>

"THIS IS AN EXACT COPY OF
 THE ORIGINAL DOCUMENT"

BY NJK DATE 12/22/09

FedEx® US Airbill

FedEx
Tracking
Number

8694 2057 8384

1 From

Date 12/21/09

Sender's
Name

Darrell Kavanaugh Phone 706 546-5839

Company

West White Stone / Delta Utilities

Address

200 Phoenix Road

City

Athens State GA ZIP 30602

2 Your Internal Billing Reference

3 To

Recipient's
Name

Daniel Wright Phone 914 272-1039

Company

MPI Research Labs

Recipient's
Address

30493 Research Drive

We cannot deliver to P.O. boxes or P.O. ZIP codes.

Address

State College State PA ZIP 16801

To request a package be held at a specific FedEx location, print FedEx address here.



8694 2057 8384

Recipient's Copy

4a Express Package Service

☒ FedEx Priority Overnight
Next business morning, ¹ Friday
shipments will be delivered on Monday
unless SATURDAY Delivery is selected.

☐ FedEx 2Day
Second business day, ² Thursday
shipments will be delivered on Monday
unless SATURDAY Delivery is selected.

☐ FedEx Express Saver
Third business day, ³ Thursday
shipments will be delivered on Monday
unless SATURDAY Delivery is selected.

4b Express Freight Service

☐ FedEx 1Day Freight⁴
Next business day, ⁵ Friday
shipments will be delivered on Monday
unless SATURDAY Delivery is selected.

☐ FedEx 2Day Freight⁴
Second business day, ⁶ Thursday
shipments will be delivered on Monday
unless SATURDAY Delivery is selected.

☐ FedEx 3Day Freight⁴
Third business day, ⁷ Saturday
shipments will be delivered on Monday
unless SATURDAY Delivery is selected.

* Call for Confirmation

5 Packaging

☐ FedEx Envelope⁸

☐ FedEx Pak⁸
Includes FedEx Small Pak,
FedEx Large Pak, and FedEx Sturdy Pak.

☐ FedEx Box

☒ Other⁹

6 Special Handling

☐ SATURDAY Delivery¹⁰
Not available for:
FedEx Standard Overnight,
FedEx First Overnight, FedEx Express
Saver, or FedEx 3Day Freight.

☐ HOLD Weekday
at FedEx Location
Not available for:
FedEx First Overnight,
FedEx Express Saver, or FedEx 3Day Freight.

☐ HOLD Saturday
at FedEx Location
Available ONLY for FedEx Priority
Overnight and FedEx 2Day
to select locations.

Does this shipment contain dangerous goods?
One box must be checked.

☒ No ☐ Yes ☐ Yes
per attached
Shipper's Declaration
Dangerous goods (including dry ice) cannot be shipped in FedEx packaging.

☐ Dry Ice
Dry Ice, 3.11 Liters 1 x 2.5 Liters

☐ Cargo Aircraft Only

Obtain Recip.
Acct. No. ☐

7 Payment Bill to: ☐ Enter FedEx Acct. No. or Credit Card No. below. ☐ Recipient ☐ Third Party ☐ Credit Card ☐ Cash/Check

Sender
Acct. No. in
bill, we
will bill.

Total Packages

Total Weight

Total Declared Value¹¹

Your liability is limited to \$100 unless you declare a higher value. See back for details.

Credit Card Auth.

8 Residential Delivery Signature Options

If you require a signature, check Direct or Indirect.

No Signature Required ☒ Direct Signature ☐ Indirect Signature

For one or more of the following, a signature is required for delivery. For signature, see signature.

Rev. Date 10/06-PAT/15/2011 © 1994-2008 FedEx/PRINTED IN U.S.A. SRY

THIS IS AN EXACT COPY OF THE ORIGINAL DOCUMENT

BY MR DATE 12/21/09



TEMPORARY SAMPLE STORAGE FORM

To be completed during ExyLIMS Login

Project #: 75196

Login #: L19897

Initials / Date: MSK 12/21/09

One form to be completed for each package

Date / Time Received: 12/22/09 1133

Received By: M. K. L.

Shipper: F&E

Shipper Package ID: 8694 2057 8384

Temperature (deg C) / Thermometer ID: -79.0° 1N0000448

Temperature Control Method: Dry Ice

Temporary Storage Location: Walker Freezer 11

Condition of sample(s):

- ☒ Good – Package and contents uncompromised
☐ Fair – Package damaged / contents uncompromised
☐ Poor – Package and contents compromised

Notes:

B



Summary of Fluorochemical Residues in Muscle Samples

Sample ID	PFOA Perfluorooctanoic Acid	PFOS Perfluorooctanesulfonate
	Analyte Found (ng/g, ppb)	Analyte Found (ng/g, ppb)
Wild Turkey # 2 4yr male-muscle	ND	82.6
Wild Turkey # 2 4yr male-muscle*	NQ	89.6
Wild Turkey # 5 1yr male-muscle	NQ	176
Wild Turkey # 5 1yr male-muscle*	NQ	193

*Laboratory Duplicate

ND = Not detected = Response is below the LOD of 1.0 ng/g (ppb).

NQ = Not quantifiable = Response is between the LOD and the LOQ of 10 ng/g (ppb).



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Analytical Report

Summary of Fluorochemical Residues in Liver Samples

Sample ID	PFOA	PFOS
	Perfluorooctanoic Acid	Perfluorooctanesulfonate
	Analyte Found (ng/g, ppb)	Analyte Found (ng/g, ppb)
Wild Turkey # 2 4yr male-liver	NQ	1560
Wild Turkey # 2 4yr male-liver*	NQ	1580
Wild Turkey # 5 1yr male-liver	12.8	2140
Wild Turkey # 5 1yr male-liver*	12.3	1970

*Laboratory Duplicate

ND = Not detected = Response is below the LOD of 1.0 ng/g (ppb).

NQ = Not quantifiable = Response is between the LOD and the LOQ of 10 ng/g (ppb).

Recovery Summary of Fluorochemical Residues in Muscle Samples

Sample Description	Amount Spiked (ng/g)	Amt Found In Sample (ng/g)	PFOA		Amt Found in Sample (ng/g)	PFOS	
			Amount Recovered (ng/g)	Recovery (%)		Amount Recovered (ng/g)	Recovery (%)
LCS A (Data set 020910A) 10 ng/g	10	NQ	9.77	98	ND	8.05	81
LCS B (Data set 020910A) 50 ng/g	50	NQ	49.6	99	ND	54.3	109
LCS A (Data set 021210A) 200 ng/g	200	N/A	N/A	N/A	ND	205	103
LCS B (Data set 021210A) 200 ng/g	200	N/A	N/A	N/A	ND	171	86
Wild Turkey # 2 4yr male-muscle (L19897-1 Spk C, 50 ng/g Lab Spike)	50	ND	49.5	99	N/A	N/A	N/A
Wild Turkey # 5 1yr male-muscle (L19897-3 Spk D, 50 ng/g Lab Spike)	50	ND	50.6	101	176	212	72
Wild Turkey # 2 4yr male-muscle (L19897-1 Spk C, 200 ng/g Lab Spike)	200	N/A	N/A	N/A	82.6	238	78

ND = Not detected = Response is below the LOD of 1.0 ng/g.

NQ = Not quantifiable = Response is between the LOD and the LOQ of 10 ng/g.

Recovery Summary of Fluorochemical Residues in Liver Samples

Sample Description	Amount Spiked (ng/g)	Amt Found in Sample (ng/g)	PFOA		Amt Found in Sample (ng/g)	PFOS	
			Amount Recovered (ng/g)	Recovery (%)		Amount Recovered (ng/g)	Recovery (%)
LCS A (Data set 020910B) 10 ng/g	10	ND	10.3	103	ND	7.64	76
LCS B (Data set 020910B) 50 ng/g	50	ND	49.6	99	ND	65.8	132
LCS A (Data set 021210B) 2000 ng/g	2000	N/A	N/A	N/A	ND	1880	94
LCS B (Data set 021210B) 2000 ng/g	2000	N/A	N/A	N/A	ND	2080	104
Wild Turkey # 2 4yr male-liver (L19897-2 Spk C, 50 ng/g Lab Spike)	50	NQ	58.4	117	1560	**	**
Wild Turkey # 5 1yr male-liver (L19897-4 Spk D, 50 ng/g Lab Spike)	50	12.8	63.3	101	2140	**	**
Wild Turkey # 2 4yr male-liver (L19897-2 Spk C, 2000 ng/g Lab Spike)	2000	N/A	N/A	N/A	1560	3870	116
Wild Turkey # 5 1yr male-liver (L19897-4 Spk D, 2000 ng/g Lab Spike)	2000	N/A	N/A	N/A	2140	4110	99

ND = Not detected = Response is below the LOD of 1.0 ng/g.

NQ = Not quantifiable = Response is between the LOD and the LOQ of 10 ng/g.